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INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the methods in the "USEPA Contract Laboratory Program Statement of Work Pages for Organics Analysis Low Concentration Water OLC0**3.2**," December 2000. The validation methods and actions discussed in this document are based on the requirements set forth in the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," June 2001. This document attempts to cover technical as well as contractual problems specific to each fraction; however, situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

In addition to technical requirements, contractual requirements are also covered in this document. While it is important that instances of contract non-compliance be addressed in the Data Assessment, the technical criteria are always used to qualify the analytical data.

Summary

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are as follows:

Data Qualifiers

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for

which there is presumptive evidence to make a "tentative identification."

NJ - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

Lab Qualifiers:

D - The positive value is the result of an analysis at a secondary dilution factor.

B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.

E - The concentration of this analyte exceeds the calibration range of the instrument.

P - Pesticide/Aroclor target analytes when the % Difference between the analyte concentrations obtained from the two dissimilar GC columns is greater than 25%.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance.

Reviewer Qualifications:

Data reviewers must possess a working knowledge of the USEPA Statement of Work OLC03.2 and National Functional Guidelines mentioned above.

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_______________________**LAB:**_________________________________

SITE NAME:_________________________**SDG No(s).:**__________________________

1.0 Chain of Custody and Sampling Trip Reports

1.1 Are the Traffic Reports/Chain-of-Custody Records present for all samples? [ ]

ACTION: If no contact RSCC, or the TOPO to obtain replacement of missing or illegible copies from the lab.

1.2 Is the Sampling Trip Report present for all samples and all fractions? [ ]

ACTION: If no, contact either RSCC or ask the TOPO to obtain the necessary information from the prime contractor.

2.0 Data Completeness and Deliverables

2.1 Have any missing deliverables been received and added to the data package? [ ]

ACTION: Contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the lab. If lab cannot provide them, note the effect on the review of the data package in the Contract Problems/Non-compliance section of the Data Assessment.

2.2 Was CLASS CCS checklist included with the package? [ ]

2.3 Are there any discrepancies between the Traffic Reports/Chain-of-Custody Records, Sampling Trip Report and Sample Tags? [ ]

	YES	NO	N/A

ACTION: If yes, contact the TOPO to obtain an explanation or resubmittal of any missing deliverables from the laboratory.

3.0 Cover Letter SDG Narrative

	YES	NO	N/A
3.1 Is the SDG Narrative or Cover Letter Present?	[ ]	___	___
3.2 Are case number, SDG number and contract number contained in the SDG Narrative or cover letter (see SOW, Exhibit B, section 2.5.1)? EPA sample numbers in the SDG, detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing the samples? Corrective action taken?	[ ]	___	___
3.3 Does the Narrative contain the following information (see SOW, page B-12, section 2.5.1):			
VOA: description or trap and column(s) used during sample analyses?	[ ]	___	___
BNA: description of column(s) used during sample analyses?	[ ]	___	___
PEST: description of columns used during sample analyses?	[ ]	___	___

NOTE: As stated in the SOW, page D-11/PEST, section 6.10.1.3.7, packed columns cannot be used.

	YES	NO	N/A
3.4 Does the narrative, VOA and BNA sections, contain a list of all TICs identified as alkanes and their estimated concentrations?	[ ]	___	___
3.5 Is the temperature indicator bottle present in the cooler? If not, did the Laboratory document in the SDG Narrative the alternative technique used to determine the cooler temperature?(Exhibit A/ p. A-7 sec. 4.2.1.2.3.3)	[ ]	___	___

	YES	NO	N/A
3.6 Does the narrative contain a list of the pH values determined for each water sample submitted for volatiles analysis (SOW, page B-13, section 2.5.1.2)?	[ ]	___	___
3.7 Does the Case Narrative contain the "verbatim" statement as required on page B-12, section 2.5.1 of the SOW?	[ ]	___	___

ACTION: If "No", to any question in this section, contact the TOPO to obtain necessary resubmittals. If the information is unavailable, document under the Contract Problems/Non-Compliance section of the Data Assessment.

4.0 Data Validation Checklist

4.1 Check the package for the following (see SOW reporting requirements, section 2.1, page B-10):

	YES	NO	N/A
a. Is the package paginated in ascending order starting from the SDG narrative?	[ ]	___	___
b. Are all forms and copies legible?	[ ]	___	___
c. Is each fraction assembled in the order set forth in the SOW?	[ ]	___	___

The following checklist is divided into three parts. Part A is filled out if the data package contains any Low Concentration Volatile analyses, Part B for any Low Concentration Semivolatile analyses and Part C for Low Concentration Pesticide/Aroclors.

Does this package contain:

	YES	NO
Low Concentration Volatiles Data?	___	___
Low Concentration Semivolatiles Data?	___	___

	YES	NO	N/A
Low Concentration Pesticides/Aroclors data?	___	___	

ACTION: Complete corresponding parts of checklist.

PART A: VOA ANALYSES

1.0 Sample Conditions/Problems

1.1 Do the Traffic Reports/Chain-of-Custody Records, Sampling Trip Report or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special circumstances affecting the quality of the data? ___ [ ] ___

ACTION: If samples were not iced or the ice was melted upon arrival at the laboratory and the temperature of the cooler was > 10° C, then flag all positive results with a "J" and all non-detects "UJ".

ACTION: If both VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

2.0 Holding Times

2.1 Have any VOA technical holding times, determined from date of collection to date of analysis, been exceeded? ___ [ ] ___

Technical Holding Times: The technical holding time criterion for water samples is 14 days from sample collection provided that samples are acid-preserved to pH 2 or below, and that they are stored in 4°C ± 2°C. If uncertain about preservation, notify the TOPO to contact the sampler and determine whether or not samples were preserved.

ACTION: List sampling, VTSR, analysis dates and preservation for samples which missed holding time in the table below.

Table of Holding Time Violations
(See Chain-of-Custody Records)

Sample ID	Was Sample Preserved?	Date Sampled	Date Lab Received	Date Analyzed
____________	____________	____________	____________	____________
____________	____________	____________	____________	____________
____________	____________	____________	____________	____________
____________	____________	____________	____________	____________

ACTION: Qualify sample results using preservation and technical holding time information as follows:

a. If there is no evidence that the samples were properly preserved, but were analyzed within the technical holding time (14 days from sample collection), qualify all positive results for non-halogenated compounds (including ketones and aromatics) with "J" and non-detects "R".

b. If there is no evidence that the samples were properly preserved, but were analyzed within 14 days from sample collection, qualify all positive results for halogenated compounds with "J" and non-detects "UJ".

c. If there is no evidence that the samples were properly preserved, and the samples were analyzed beyond 14 days from sample collection, qualify positive results for all volatile compounds with "J" and non-detects "R".

d. If the samples were properly preserved, but were analyzed outside of the technical holding time (14 days from sample collection), qualify positive results for all volatile compounds with "J" and non-detects "R".

NOTE: <u>Contractual Holding Times</u>: Sample must be analyzed within 10 days from validated time of sample receipt (VTSR) at the laboratory.

3.0 <u>Deuterated Monitoring Compound (DMC) Recovery (Form II LCV)</u>

3.1 Are the Volatile SMC Recovery Summaries (Form II LCV-1 and LCV-2) present? [ ] ___ ___

ACTION: Call the TOPO to obtain an explanation/resubmittal from the lab. If missing deliverables are unavailable, document the effect in the Data Assessment.

3.2 Were outliers marked correctly with an asterisk? [ ] ___ ___

ACTION: Circle all outliers in red.

3.3 Were more than three of the fourteen (14) Deuterated Monitoring Compounds (DMC's) recoveries outside their corresponding limits? ___ [ ] ___

If yes, were samples re-analyzed? [ ] ___ ___

Were method blanks re-analyzed? [ ] ___ ___

ACTION: If any DMC is outside the required limits (see Table below), qualify their associated target compounds (See Table below) as follows:

<u>VOLATILE DMC AND THEIR ASSOCIATED TARGET COMPOUNDS</u>

Chloroethane-d5 Dichlorodifluoromethane Chloromethane Bromomethane Chloroethane Carbon Disulfide	**1,2-Dichloropropane-d6** Cyclohexane Methylcyclohexane 1,2-Dichloropropane Bromodichloromethane	**1,2-Dichlorobenzene-d4** Chlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene 1,2-Dichlorobenzene 1,2,4-Trichlorobenzene 1,2,3-Trichlorobenzene
Bromoform-d Dibromochloromethane 1,2-Dibromoethane Bromoform	**trans-1,3-Dichloropropene-d4** cis-1,3-Dichloropropene trans-1,3-Dichloropropene 1,1,2-Trichloroethane	**Chloroform-d** 1,1-Dichloroethane Bromochloromethane Chloroform
2-Butanone-d5 Acetone 2-butanone	**1,1-dichloroethene-d2** trans-1,2-Dichloroethene cis-1,2-Dichloroethene	**2-Hexanone-d5** 4-Methyl-2-pentanone 2-Hexanone
Vinyl Chloride-d3 Vinyl Chloride	**Benzene-d6** Benzene	**1,1,2,2-Tetrachloroethane-d2** 1,1,2,2-Tetrachloroethane 1,2-Dibromo-3-chloropropane

1,2-Dichloroethane-d4	**Toluene-d8**	
Trichlorofluoromethane 1,1-Dichloroethene 1,1,2-Trichloro-1,2,2-trifluoroethane Methyl Acetate Methylene Chloride Methyl tert-Butyl Ether Carbon Tetrachloride 1,2-Dichloroethane 1,1,1-Trichloroethane	Trichloroethene Toluene Tetrachloroethene Ethylbenzene Xylenes (total) Styrene Isopropylbenzene	

VOLATILE DEUTERATED MONITORING COMPOUND RECOVERY LIMITS

DMC	%RECOVERY LIMITS	DMC	%RECOVERY LIMITS
Vinyl Chloride-d3	49-138	1,2-Dichloropropane-d6	84-123
Chloroethane-d5	60-126	Toluene-d8	77-120
DMC	%RECOVERY LIMITS	DMC	%RECOVERY LIMITS
1,1-Dichloroethene-d2	65-130	trans-1,3-Dichloropropane-d4	80-128
2-Butanone-d5	42-171	2-Hexanone-d5	37-169
Chloroform-d	80-123	Bromoform-d	76-135
1,2-Dichloroethane-d4	78-129	1,1,2,2-Tetrachloroethane-d2	75-131
Benzene-d6	78-121	1,2-Dichlorobenzene-d4	50-150

1. For any recovery greater than the upper limit:

   a. Qualify "J" all positive associated target compounds.
   b. Do not qualify associated non-detects.

2. For any recovery greater than or equal to 20%, but less than the lower limit:

   a. Qualify "J" all positive associated target compounds.
   b. Qualify "UJ" associated non-detects.

3. For any recovery less than 20%:

   a. Qualify "J" all positive associated target compounds.
   b. Qualify "R" all associated non-detects.

NOTE: Up tp three (3) DMC's per sample may fail to meet the recovery limits. (SOW OLC03.2, sec. 11.4.4, p. D-41/VOA)
As per SOW, any sample which has more than 3 DMC's outside the limits, it must be reanalyzed (sec. 11.5.1 p. d-42/VOA).

ACTION: Note in the Data Assessment under Contract Problems/ Non-Compliance if the Lab did not perform reanalysis.

3.4 Are there any transcription/calculation errors between raw data and form II? ___ [ ] ___

ACTION: If large errors exist, ask the TOPO to obtain an explanation/resubmittal from the lab, make any necessary corrections and note errors in the data assessment.

4.0 <u>Matrix Spike/Matrix Spike Duplicate Recovery (Form III LCV)</u>

4.1 Is the MS/MSD Recovery Form (Form III LCV) present? [ ] ___ ___

4.2 Was the MS/MSD analyzed at the required frequency (once per SDG, or every 20 samples,

YES NO N/A

whichever is more frequent) for the Low Concentration VOA method? [ ] ___ ___

ACTION: If any MS/MSD data are missing, take action as specified in section 3.1 above.

ACTION: No action is taken on MS/MSD data <u>alone.</u> However, Using professional judgement, the Validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

5.0 <u>Method Blanks (Form IV LCV)</u>

5.1 Is the Volatile Method Blank Summary (Form IV LCV) present? [ ] ___ ___

5.2 <u>Frequency of Analysis</u>: For the analysis of Low Concentration VOA TCL compounds, has a method blank been analyzed for each SDG or every 20 samples, whichever is more frequent? [ ] ___ ___

5.3 Has a VOA method blank been analyzed at least once every twelve hours for each GC/MS system used? [ ] ___ ___

5.4 Was a VOA instrument blank analyzed after each sample/dilution which contained a target compound at a concentration > 25 :g/R, and ketones > 125 :g/R (see SOW, page D-44/VOA, section 12.1.1.3)? [ ] ___ ___

ACTION: If any method/instrument blank data are missing, notify the TOPO to obtain resubmittals or an explanation from the lab. If method blank data are unavailable, the reviewer may use professional judgement, or substitute field blank or trip blank data for missing method blank data.

If an instrument blank was not analyzed after a sample containing > 25 :g/R, (ketones > 125 :g/R) inspect the sample chromatogram acquired immediately after this sample

for possible carryover. Use professional judgement to determine if carryover occurred and qualify analyte(s) accordingly.

5.5 Was a storage blank analyzed once per SDG after all the samples were analyzed? [ ] ___ ___

ACTION: If storage blank data is missing, contact the TOPO to obtain any missing deliverables from the laboratory. If unavailable, note in the Contract Problems/Non-Compliance section of the Data Assessment.

5.6 The validator should verify that the correct identification scheme for EPA blanks was used. (See SOW page B-30, section 3.3.7.3 for more information.)

Was the correct identification scheme used for all Low Concentration VOA blanks? [ ] ___ ___

ACTION: Contact the TOPO to obtain corrections from the lab, or make the necessary corrections. Document in the "Contract Problems/Non-Compliance section of the Data Assessment all corrections made by the validator.

5.7 Chromatography: review the blank raw data - chromatograms (RICs), quant. reports, data system printouts and spectra.

Also compare the storage blank raw data with the method blank. Determine if contamination in the storage blank is also present in the method blank.

Is the chromatographic performance (baseline stability) for each instrument acceptable for Low Concentration VOAs? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

5.8 Are all detected hits for target compounds in method, instrument and storage blanks less than the CRQL for that analyte? [ ] ___ ___

Exception: Acetone and 2-butanone must be less than 2X times the CRQL, and Methylene Chloride and Cyclohexane must be less than 10X times its CRQL.

ACTION: If no, an explanation and laboratory's corrective actions must be addressed in the case narrative. If the narrative contains no explanation, then make a note in the Contract Problems/Non-Compliance section of the Data Assessment.

6.0 Contamination

NOTE: "Water blanks", "drill blanks", and distilled water blanks" are validated like any other sample, and are not used to qualify data. Do not confuse them with the other QC blanks discussed below.

6.1 Does the storage blank contain positive results (TCL and/or TICs) for Low Concentration VOAs? ___ [ ] ___

ACTION: If the storage blank contains target compounds at a concentration greater than the CRQL, positive sample results for those compounds should be flagged "J". If gross contamination occurred positive sample results for that compound may be rejected (R).

6.2 Do any method/reagent/instrument blanks contain positive results (including TICs) for Low Concentration VOAs? When applied as described in the table below, the contaminant concentration in these blanks are multiplied by the sample dilution factor. ___ [ ] ___

NOTE: Contaminated instrument blanks are unacceptable under this SOW (see page D-46/VOA, section 12.1.6.2).

ACTION: Document in the Data Assessment under Contract Problems/Non-Compliance if a contaminated instrument blank was submitted.

ACTION: Sample analysis results after the high concentration sample must be evaluated for carryover. Sample must meet the maximum carryover criteria as listed in SOW sec. 11.4.9.2, p. D-42/VOA.("the sample must not contain a concentration above the CRQL for the target compounds that exceeded the limit in the contaminated sample.")

6.3 Do any field/trip/rinse blanks have positive Low Concentration VOA results (including TICs)? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Trip blanks are used to qualify only those samples with which they were shipped. Blanks may not be qualified because of contamination in another blank. Field blanks & trip blanks must be qualified for system monitoring compound, instrument performance criteria, spectral or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If any blanks are grossly contaminated, all associated sample data should be qualified unusable (R).

For:	**Flag sample result with a "U" when:**	**Report CRQL & qualify "U" when:**	**No qualification is needed when:**
Methylene Chloride **Cyclohexane**	Sample conc. is > CRQL, but < 10x blank value.	Sample conc. is < CRQL and < 10x blank value.	Sample conc. is > CRQL and > 10x blank value.
Acetone **2-Butanone**	Sample conc. is > CRQL, but < 2x blank value.	Sample conc. is < CRQL and < 2x blank value.	Sample conc. is > CRQL and > 2x blank value.

Other contami- nants	Sample conc. is > CRQL, but < 1x blank value.	Sample conc. is < CRQL and < 1x blank value.	Sample conc. is > CRQL and > 1x blank value.

NOTE: Analytes qualified "U" for blank contamination are treated as "hits" when qualifying for calibration criteria.

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R" (unusable).

6.4 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: Note in data assessment that there is no associated field/rinse/equipment blank.

<u>Exception</u>: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Instrument Performance Check (Form V-LCV)

7.1 Are the GC/MS Instrument Performance Check Forms (Form V-LCV) present for Bromofluorobenzene (BFB)? [ ] ___ ___

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift? [ ] ___ ___

7.3 Has an instrument performance compound been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

ACTION: List date, time, instrument ID and sample analyses for which associated GC/MS tuning data are missing.

DATE	**TIME**	**INSTRUMENT ID**	**SAMPLE NUMBERS**
________	________	__________________	_______________
________	________	__________________	_______________
________	________	__________________	_______________

ACTION: Notify the TOPO to obtain missing data from the lab. If the lab cannot provide missing data, reject (R) all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundances been normalized to m/z 95 (see SOW, page D-24/VOA)? [ ] ___ ___

NOTE: All ion abundance ratios must be normalized to m/z 95, the nominal base peak, even though the ion abundance of m/z 174 may be up to 120% that of m/z 95.

ACTION: If mass assignment is in error, qualify all associated data as unusable (R).

7.5 Have the ion abundance criteria been met for each instrument used? [ ] ___ ___

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, professional Judgement may be applied to determine to what extent the data may be utilized.

7.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) ___ [ ] ___

7.7 Is the number of significant figures for the reported relative abundances consistent with the number given in the ion abundance criteria column on Form V LCV? [ ] ___ ___

ACTION: If large errors exist, take action as specified in section 3.1 above.

7.8 Is the spectrum of the mass calibration compound acceptable? [ ] ___ ___

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

8.0 Target Compound List (TCL) Analytes (Form I LCV)

8.1 Are the Organic Analysis Data Sheets (Form I LCV) present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate? [ ] ___ ___

b. Laboratory Control/MS/MSD samples? [ ] ___ ___

c. Blanks? [ ] ___ ___

8.2 Are the VOA Reconstructed Ion Chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following:

a. Samples and/or fractions as appropriate? [ ] ___ ___

b. Laboratory Control/MS/MSD samples? [ ] ___ ___

c. Blanks? [ ] ___ ___

ACTION: If any data are missing, take action specified in 3.1 above.

	YES	NO	N/A
8.**3** Is chromatographic performance acceptable with respect to:			
Baseline stability?	[ ]	___	___
Resolution?	[ ]	___	___
Peak shape?	[ ]	___	___
Full-scale graph (attenuation)?	[ ]	___	___
Other: _________________________?	[ ]	___	___

ACTION: Use professional judgement to determine the acceptability of the data.

8.4 Are lab-generated standard mass spectra of the identified VOA compounds present for each sample?	[ ]	___	___

ACTION: If any mass spectra are missing, take action as specified in 3.1 above. If lab does not generate their own standard spectra, make note under the "Contract Problems/Non-Compliance" section of the Data Assessment. If spectra are unavailable reject “R” the reported results.

8.5 Is the RRT of each reported compound within ±0.06 RRT units of the standard RRT in the continuing calibration?	[ ]	___	___
8.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	[ ]	___	___
8.7 Do sample and standard relative ion intensities agree to within ±20%?	[ ]	___	___

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be

rejected (R) flagged "N" (presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in sections 8.4-8.7 above.

ACTION: When sample carry-over is suspected, use professional judgement to determine if instrument cross-contamination has affected positive compound identifications.

9.0 Tentatively Identified Compounds (TIC)

9.1 Are all Tentatively Identified Compound Forms (Form I LCV-TIC) present? Do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? [ ] ___ ___

9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate? [ ] ___ ___

b. Blanks? [ ] ___ ___

b. Are Alkanes listed in/or part of the Case Narrative? [ ] ___ ___

ACTION: If any TIC data are missing, take action specified in 3.1 above.

ACTION: Add "JN" qualifier to all chemically named TICs if missing.

9.3 Are any target compounds (from any fraction) listed as TICs? (Example: 1,2-dimethylbenzene is xylene - a VOA target analyte - and should not be reported as a TIC.) ___ [ ] ___

ACTION: Flag with "R" only target compound detected in another fraction. (Except blank contamination)

9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? [ ] ___ ___

9.5 Do TIC and "best match" standard relative ion intensities agree within $\pm$20%? [ ] ___ ___

ACTION: Use professional judgement to determine the acceptability of TIC identifications. If it is determined that an incorrect identification was made, change its identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is detected in a sample and is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable (R). (I.e., common lab contaminants such as CO_2 - M/E 44, Siloxanes - M/E 73, hexane, Aldol condensation products, solvent preservatives, and related by-products. See the National Functional Guidelines June 2001, pp. 34-35 for further guidance.)

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? (Check at least two positive values. Verify that the correct internal standards, quantitation ions, and RRFs were used to calculate Form I results.) ___ [ ] ___

10.2 Are the CRQLs adjusted to reflect sample dilutions? [ ] ___ ___

ACTION: If errors are large, take action as specified in section 3.1 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQLs data from the diluted sample). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and its corresponding value on the original Form I and substituting the data from the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's not to be used, including any in the data summary package.

11.0 <u>Standards Data (GC/MS)</u>

11.1 Are the reconstructed ion chromatograms, and data system printouts (quant. reports) present for each initial and continuing calibration? [ ] ___ ___

ACTION: If any calibration standard data are missing, take action specified in section 3.1 above.

12.0 <u>GC/MS Initial Calibration (Form VI)</u>

12.1 Are the Initial Calibration Forms (Form VI LCV) present and complete for the volatile fraction at concentrations of 0.5, 1, 5, 10, and 25 :g/R? [ ] ___ ___

ACTION: If any Initial Calibration forms are missing, take action as specified in section 3.1 above.

12.2 Are response factors stable for VOA's over the concentration range of the calibration (e.g., %RSD # 30.0, ≤50 for poor performers)? [ ] ___ ___

ACTION: Circle all outliers in red.

NOTE: There are fourteen (14) compounds (see Table below) which are poor performers. The RRF for these compounds must be greater than or equal to 0.01. The %RSD must be less than or equal to 50%.

VOLATILE COMPOUNDS WITH POOR RESPONSE

Volatile Compounds	
Acetone	1,2-Dichloropropane
2-Butanone	1,2-Dibromo-3-chloropropane
Carbon Disulfide	4-Methyl-2-pentanone
Chloroethane	2-Hexanone
Chloromethane	1,2-Dichloropropane-d6 (DMC)
Cyclohexane	2-Hexanone-d5 (DMC)
Chloroethane-d5 (DMC)	2-Butanone-d5 (DMC)

NOTE: Although 20 Low Conc. VOA compounds have no maximum %RSD and require only minimal $\overline{RRF}$ performance (see Table D-2, page D-53/VOA), the technical acceptance criteria are the same for all analytes.

ACTION: If %RSD > 30.0%, or > 50.0% for the poor performers, qualify associated positive results for that analyte "J" (estimated) and non-detects using professional judgement. If %RSD is > 90, flag all non-detects for that analyte "R" (unusable) and positive hits "J".

NOTE: Analytes previously qualified "U" for blank contamination are still treated as "hits" when qualifying for initial calibration criteria.

12.3 Are any $\overline{RRF}$s < 0.05 or < 0.01 for poor performers? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: If any $\overline{RRF}$ values are < 0.05 or < 0.01 for poor performers, qualify associated non-detects unusable (R) and associated positive results estimated (J).

NOTE: <u>Contract Requirements</u>: The SOW allows up to two of the <u>required</u> analytes (see compounds marked with a "*" on Form VI and Table D-2, page D-53/VOA) to fail contractual %RSD and RRF criteria, provided the %RSD is # 40.0 and RRF $ 0.010.

ACTION: If more than two of the required analytes failed %RSD or RRF criteria, document in the Data Assessment under Contract Problems/Non-Compliance.

12.4 Are there any transcription/calculation errors in the reporting of RRFs, RRFs or %RSD values? (Check at least 2 values, but if errors are found, check more.) ___ [ ] ___

ACTION: Circle errors in red.

ACTION: If errors are large, contact the TOPO to obtain an explanation/resubmittal from the lab, document in the Data Assessment under Contract Problems/Non-Compliance.

13.0 <u>GC/MS Continuing Calibration (Form VII LCV)</u>

13.1 Are the Continuing Calibration Forms (Form VII LCV) present and complete for the volatile fraction? [ ] ___ ___

13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of every sample analysis, ask the TOPO to obtain explanation/resubmittal from the laboratory. If continuing calibration data are unavailable, flag all associated sample data as unusable (R).

ACTION: List below all sample analyses that were not within twelve hours of the previous continuing calibration analysis.

____

13.3 Do any volatile compounds have a % Difference (%D) between the initial RRF and continuing RRF which exceeds the ± 30% , or ± 50% for the poor performers criteria? ___ [ ] ___

ACTION: Circle all outliers in red.

NOTE: Although 20 Low Conc. VOA compounds have no maximum %D and require only minimal RRF performance (see Table D-2, page D-53/VOA), the technical acceptance criteria are the same for all analytes.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When % D is above 90%, reject all non-detects for that analyte as unusable (R) and qualify positive results "J".

13.4 Do any volatile compounds have a RRF < 0.05 or < 0.01 for the poor performers? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: If the RRF < 0.05, or < 0.01 for poor performers qualify associated positive results as estimated (J) and associated non-detects unusable (R).

NOTE: Contract Requirements: The SOW allows up to two of the required analytes (see compounds marked with a "*" on Form VI, or Table D-2, page D-53/VOA) to fail %D or RRF criteria, provided %D is within ±40.0 and RRF $ 0.010.

ACTION: Document in the Data Assessment under Contract Problems/Non-Compliance if more than two of the required analytes failed the above acceptance criteria.

13.5 Are there any transcription/calculation errors in the reporting of RRFs, or %D between initial RRFs and continuing RRFs? (Check at least two values but if errors are found, check more.) ___ [ ] ___

ACTION: Circle errors with red pencil.

ACTION: If errors are large, notify the TOPO to obtain explanation/resubmittals from the lab. Document errors in the Contract Problems/Non-Compliance section of the Data Assessment.

14.0 Internal Standard (Form VIII LCV)

14.1 Are the internal standard areas (Form VIII LCV) of every sample and blank within the upper and lower limits (± 40%) for each continuing calibration? [ ] ___ ___

If no, was the sample reanalyzed? [ ] ___ ___

ACTION: 1. Circle all outliers with red pencil.

2. List all the outliers below.

Sample #	Int. Std.	Area	Lower Limit	Upper Limit

(Attach additional sheets if necessary, or attach copies of Form VIIIs.)

ACTION: 1. If the internal standard area count is outside the **upper** limit, flag with "J" all positive results quantitated with this internal standard.

2. Do not qualify non-detects when associated IS area counts are > +40%.

3. If the IS area is less than the lower limit (-40%), qualify “J” all positive results quantitated with this Internal Standard. Qualify “R” all non-detects.

INTERNAL STANDARDS ACTIONS FOR VOLATILES

CRITERIA	ACTION	
	Detected Associated Compounds	Non-detected Associated Compounds
Area counts > 40% of 12-hour standard	"J"	No Action
Area counts < 40% of 12-hour	"J"	"R"

14.2 Are the retention times of the internal standards within ±20 seconds of the associated calibration standard? [ ] ___ ___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 20 seconds.

NOTE: Contract Requirements: The SOW (section 11.5.1 page D-41/VOA) states that any sample which fails the acceptance criteria for IS response must be reanalyzed.

ACTION: Document in the Data Assessment under Contract Problems/Non-Compliance any sample(s) which failed the above IS acceptance criteria.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for Low Concentration VOA analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between duplicate results must be addressed in the reviewer narrative. If large

differences exist, contact the TOPO to confirm identification of field duplicates with the sampler.

PART B: BNA ANALYSES

1.0 Sample Conditions/Problems

1.1 Do the Traffic Reports/Chain-of-Custody records or SDG Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data? ___ [ ] ___

ACTION: If samples were not iced or the ice west melted upon arrival at the laboratory and the temperature of the cooler was > 10° C, then flag all positive results with a "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any Low Concentration semivolatile technical holding times, determined from the date of collection to date of extraction, been exceeded? ___ [ ] ___

Technical Holding Time: Continuous liquid-liquid extraction of BNA samples must begin within seven days of the date of collection. Extracts must be analyzed within 40 days from the extraction date.

Table of Holding Time Violations
(See Chain-of-Custody records)

Sample ID	Date Sampled	Date Lab Received	Date Extracted	Date Analyzed

ACTION: If technical holding times were exceeded, flag all positive results as estimated (J) and sample quantitation limits as estimated (UJ), and document in the narrative that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon reanalysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J" but the reviewer may determine that non-detect data are unusable (R). If holding times were exceeded by more than 28 days, qualify all non-detects unusable (R).

NOTE: Contractual Holding Times: Extraction of water samples must begin within 5 days VTSR. All laboratory extracts must be analyzed within 40 days of the VTSR.

ACTION: If contractual holding times were exceeded, document in the Data Assessment under Contract Problems/Non-Compliance.

NOTE: The data reviewer must note in the Data Assessment whether or not technical and contractual holding times were met.

3.0 Deuterated Monitoring Compound Recovery (Form II LCSV)

3.1 Are the Low Concentration Semivolatile Deuterated Monitoring Compound Recovery Summaries (Form II LCSV-1 and LCSV-2) present and complete for all samples? [ ] ___ ___

ACTION: Ask the TOPO to obtain explanations/resubmittals of any missing deliverables from the laboratory. If missing deliverables are unavailable, document the effect in the Data Assessment.

3.2 Were outliers marked correctly with an asterisk? [ ] ___ ___

ACTION: Circle all outliers in red.

3.3 Were more than four, two from each fraction, of the sixteen (16) Deuterated Monitoring Compounds

	YES	NO	N/A
(DMC's) recoveries outside their corresponding limits?	___	[ ]	___
If yes, were samples reanalyzed?	[ ]	___	___
Were method blanks reanalyzed?	[ ]	___	___

ACTION: If any DMC is outside the required limits(See Table below), qualify their associated target compounds (See Table below) as follows:

SEMIVOLATILE DMC AND THEIR ASSOCIATED TARGET COMPOUNDS

Phenol-d5 Benzaldehyde Phenol	**2-Chlorophenol-d4** 2-Chlorophenol	**2-Nitrophenol-d4** Isophorone 2-Nitrophenol
bis-(2-Chloroethyl)ether-d8 bis-(2-Chloroethyl)ether 2,2'-oxybis(1-Chloropropane) bis(2-Chloroethoxy)methane	**4-Methylphenol-d8** 2-Methylphenol 4-Methylphenol 2,4-Dimethylphenol	**4-Chloroaniline-d4** 4-Chloroaniline Hexachlorocyclopentadiene 3,3'-Dichlorobenzidine
Nitrobenzene-d5 Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene 2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosodiphenylamine	**2,4-Dichlorophenol-d3** 2,4-Dichlorophenol Hexachlorobutadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene Pentachlorophenol	**Dimethylphtalate-d6** Caprolactam 1,1'-Biphenyl Dimethylphthalate Diethylphthalate Di-n-butylphthalate Butylbenzylphthalate bis(2-Ethylhexyl)phthalate Di-n-octylphthalate

Fluorene-d10	**Anthracene-d10**	**Pyrene-d10**
Dibenzofuran Fluorene 4-Chlorophenyl-phenylether 4-Bromophenyl-phenylether	Hexachlorobenzene Atrazine Phenanthrene Anthracene	Fluoranthene Pyrene Benzo(a)anthracene Chrysene
Acenaphthylene-d8 Naphthalene 2-Methylnaphthalene 2-Chloronaphthalene Acenaphthylene Acenaphthene	**4-Nitrophenol-d4** 2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol 4-nitrophenol 4-Nitroaniline	**Benzo(a)pyrene-d12** Benzo(b)fluoranthene benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene
4,6-Dinitro-2-methylphenol-d2 4,6-Dinitro-2-methylphenol		

SEMIVOLATILE DEUTERATED MONITORING COMPOUND LIMITS

COMPOUND	% RECOVERY
Phenol-d5	10-110
bis-(2-Chloroethyl)ether-d8	41-94
2-Chlorophenol-d4	33-110
4-Methylphenol-d8	38-95
Nitrobenzene-d5	35-114
2-Nitrophenol-d4	40-106
2,4-Dichlorophenol-d3	42-98
4-Chloroaniline-d4	8-70

Dimethylphthalate-d6	62-102
Acenaphthylene-d8	49-98
4-Nitrophenol-d4	9-181
Fluorene-d10	50-97
4,6-Dinitro-2-methylphenol-d2	53-153
Anthracene-d10	55-116
Pyrene-d10	47-114
Benzo(a)pyrene-d12	54-120

3.5 Are there any transcription/calculation errors between raw data and Form II? ___ [ ] ___

ACTION: .If large errors exist, ask the TOPO to obtain an explanation/resubmittal from the lab, make any necessary corrections and note errors in the Data Assessment.

ACTION: 1. For any recovery greater than the upper limit:

a. Qualify “J” all positive associated target compounds
b. Do not qualify associated non-detects.

2. For any recovery less than the lower limit:

a. Qualify “J” all positive associated target compounds
b. Qualify “UJ” all non-detects if recoveries are ≥10% except for 4-Chloroaniline-d4 and 4-Nitrophenol-d4.
c. Qualify “R” all non-detects if recoveries are < 10% except for 4-Chloroaniline-d4 and 4-Nitrophenol-d4.
d. For 4-Chloroaniline-d4 and 4-Nitrophenol-d4 qualify “R” all non-detects if recoveries are less than their lower limit.

NOTE: Up to four DMC’s (two per fraction) per sample may fail to meet the recovery limits (SOW OLC03.2, sec. 11.6.4, p. D-34/SV). As per SOW, any sample that fails the technical criteria, must be reanalyzed (sec. 11.7.4 p. D-35/SV).

ACTION: Note in the Data Assessment under Contract Problems/

Non-Compliance if he Lab did not perform reanalysis.

4.0 Laboratory MS/MSD (Form III LCSV)

4.1 Is the Semivolatile MS/MSD Recovery Form (Form III LCSV) present? [ ] ___ ___

4.2 Was the MS/MSD analyzed at the required frequency (once per SDG, or every 20 samples)? [ ] ___ ___

ACTION: If any MS/MSD data are missing, take action as specified in 3.1 above.

ACTION: No action is taken on MS/MSD alone. However, using professional judgement, the Validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

5.0 Blanks (Form IV LCSV)

5.1 Is the Method Blank Summary Form (Form IV LCSV) present? [ ] ___ ___

5.2 Frequency of Analysis: For the analysis of Low Concentration semivolatile TCL compounds, has a method blank been analyzed and reported for each SDG, every 20 samples or each extraction batch, whichever is more frequent? [ ] ___ ___

5.3 Was a Low Concentration semivolatile method blank analyzed for each GC/MS system used? (See SOW page D-36/SV, section 12.1.2.2) [ ] ___ ___

ACTION: If any method blank data are missing, ask the TOPO to obtain an explanation/resubmittal from the laboratory. If method blank data is unavailable, reject (R) all associated positive results. However, the data reviewer may, based on professional judgement, substitute field blank data for missing method blank data.

5.4 The validator should verify that the correct identification scheme for EPA blanks was used. (See SOW page B-30, section 3.3.7.3 for more information.)

Was the correct identification scheme used for all Low Concentration Semivolatile blanks? [ ] ___ ___

ACTION: Contact the TOPO to obtain corrections from the lab, or make the necessary corrections. Document in the "Contract Problems/Non-Compliance section of the Data Assessment all corrections made by the validator.

5.5 Chromatography: Review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra. Is the chromatographic performance (baseline stability) acceptable for each instrument? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

5.6 Are all detected hits for target compounds less than the CRQL for that analyte in all method blanks? [ ] ___ ___

Exception: Phthalate esters must be less than five times (5X) the CRQL.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method blanks have positive results (TCL and/or TICs) for Low Concentration Semivolatiles? ___ [ ] ___

6.2 Do any field/rinse blanks have positive results for Low Concentration Semivolatiles (TCL and/or TIC)? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated with a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of

contamination in another blank. Field blanks must be qualified for surrogate, spectral, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).

NOTE: When applied as described below, the contaminant concentration in these blanks is multiplied by the sample dilution factor.

For:	Flag sample result with a "U" when:	Report CRQL & qualify "U" when:	No qualification needed when:
Common Pthalate-Esters	Sample conc. is > CRQL, but < 5x blank value.	Sample conc. is < CRQL and < 5x blank value.	Sample conc. is > CRQL and > 5x blank value.
Other Conta-minants	Sample conc. is > CRQL, but < 1x blank value.	Sample conc. is < CRQL and < 1x blank value.	Sample conc. is > CRQL and > 1x blank value.

NOTE: Analytes qualified "U" for blank contamination are still treated as "hits" when qualifying for calibration criteria.

ACTION: For TIC compounds, if the concentration in the sample is less than five times the concentration in the most contaminated associated blank, flag the sample data "R", unusable.

6.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: Note in the Data Assessment that there is no associated field/rinse/equipment blank.

<u>Exception</u>: samples taken from a drinking water tap do not have associated field blanks.

7.0 GC/MS Instrument Performance Check (Form V LCSV)

7.1 Are the GC/MS Instrument Performance Check Forms (Form V LCSV) for Decafluorotriphenylphosphine (DFTPP) present? [ ] ___ ___

7.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift? [ ] ___ ___

7.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analyses per instrument? [ ] ___ ___

ACTION: List samples, date, time and instrument ID for which no associated GC/MS tuning data are available.

SAMPLE ID	DATE	TIME	INSTRUMENT ID
___________	___________	______________	______________________
___________	___________	______________	______________________
___________	___________	______________	______________________

ACTION: If lab cannot provide missing data, reject (R) all data generated outside an acceptable twelve hour calibration interval.

7.4 Have the ion abundances been normalized to m/z 198? [ ] ___ ___

NOTE: All ion abundance ratios must be normalized to m/z 198, the nominal base peak, even though the ion abundance of m/z 442 may up to 110% that of m/z 198.

ACTION: If mass assignment is in error, flag all associated sample data as unusable (R).

7.5 Have the ion abundance criteria been met for each instrument used? [ ] ___ ___

ACTION: If ion abundance criteria are not met, professional

Judgement may be applied to determine to what extent the data may be utilized.

7.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.) ___ [ ] ___

7.7 Is the number of significant figures for the reported relative abundances consistent with the number given for each ion in the ion abundance criteria column on Form V LCSV? [ ] ___ ___

ACTION: If large errors exist, notify the TOPO to obtain an explanation/resubmittal, make necessary corrections and document effect in data assessments.

7.8 Is the spectrum of the mass calibration compound acceptable? [ ] ___ ___

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified or rejected.

8.0 Target Compound List (TCL) Analytes (Form I LCSV)

8.1 Are the Organic Analysis Data Sheets (Form I LCSV-1, 2) present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate? [ ] ___ ___

b. Laboratory Control/MS/MSD Samples? [ ] ___ ___

c. Blanks? [ ] ___ ___

8.2 Are the Low Concentration Semivolatile reconstructed ion chromatograms, the mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following:

a. Samples and/or fractions as appropriate? [ ] ___ ___

b. Laboratory Control Sample(s) and MS/MSD? [ ] ___ ___

	YES	NO	N/A
c. Blanks	[ ]	___	___

ACTION: If any data are missing, take action as specified in 3.1 above.

8.3 Is chromatographic performance acceptable with respect to:

	YES	NO	N/A
Baseline stability?	[ ]	___	___
Resolution?	[ ]	___	___
Peak shape?	[ ]	___	___
Full-scale graph (attenuation)?	[ ]	___	___
Other: _________________________?	[ ]	___	___

ACTION: Use professional judgement to determine the acceptability of the data.

	YES	NO	N/A
8.4 Are the lab-generated standard mass spectra of identified Low Concentration semivolatile compounds present for each sample?	[ ]	___	___

ACTION: If any mass spectra are missing, take action specified in 3.1 above. If lab does not generate their own standard spectra, make note in "Contract Problems/Non-Compliance". If spectra are missing, reject the reported result(s).

	YES	NO	N/A
8.5 Is the RRT of each reported compound within $\pm$0.06 RRT units of the standard RRT in the continuing calibration?	[ ]	___	___
8.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% also present in the sample mass spectrum?	[ ]	___	___
8.7 Do sample and standard relative ion intensities agree within $\pm$20%?	[ ]	___	___

ACTION: Use professional judgement to determine the acceptability of the data. If it is determined that incorrect identifications were made, all such data should be rejected (R) flagged "N" (Presumptive

evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the qualitative identification criteria listed in SOW section 11.1, page D-29/SV.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

9.0 Tentatively Identified Compounds (TIC)

9.1 Are all Tentatively Identified Compound Forms (Form I LCSV-TIC) present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier? [ ] ___ ___

9.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate? [ ] ___ ___

b. Blanks? [ ] ___ ___

ACTION: If any TIC data are missing, take action specified in 3.1 above.

ACTION: Add "JN" qualifier to all chemically named TICs.

9.3 Are any TCL compounds (from any fraction) listed as TIC compounds (example: 1,2- dimethylbenzene is xylene a VOA TCL and should not be reported as a TIC)? ___ [ ] ___

ACTION: Flag "R" only TCL compound detected in another fraction. (Except blank contamination)

9.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% also present in the sample mass spectrum? [ ] ___ ___

9.5 Do TIC and "best match" standard relative ion intensities agree within ±20%? [ ] ___ ___

ACTION: Use professional judgement to determine the acceptability of TIC identifications. If it is determined that an incorrect identification was made, change identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. In order to be positively identified, the data must comply with the criteria listed in SOW section 11.2, page D-30/SV.

Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable (R). Common lab contaminants could be solvent preservatives, such as Cyclohexene. Related by-products include Cyclohexanone, Cyclohexanol, Chlorocyclohexene and Chlorocyclohexanol. Aldol reaction products of Acetone include 4-hydroxy-4-methyl-2-pentanone, 4-methyl-2-penten--2-one, and 5,5-dimethyl-2-(5H)-furanone.

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found? ___ [ ] ___

10.2 Are the CRQLs adjusted to reflect sample dilutions? [ ] ___ ___

ACTION: If errors are large, notify the TOPO to obtain an explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I and substituting the data from the analysis of the diluted sample. Specify which Form I

is to be used, then draw a red " X" across the entire page of all Form I's that should not be used, including any in the summary package.

11.0 <u>Standards Data (GC/MS)</u>

11.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration? [ ] ___ ___

ACTION: If any calibration standard data are missing, take action specified in 3.1 above.

12.0 <u>GC/MS Initial Calibration (Form VI LCSV)</u>

12.1 Are the Initial Calibration Forms (Form VI LCSV-1 & -2) present and complete for the Low Concentration Semivolatile fraction at concentrations of 5, 10, 20, 50 and 80 ug/l? [ ] ___ ___

NOTE: Seven compounds, 2,4-Dinitrophenol, 2,4,5-Trichlorophenol 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline 4-Nitrophenol, 4,6-Dinitro-2-methylphenol, require calibration at 20, 50, 80, 100 and 120 ug/l.

ACTION: If any calibration standard forms are missing, take action specified in 3.1 above.

NOTE: There are nineteen (19) semivolatile compounds (see Table below) which are poor performers. The RRF for these compounds must be greater than or equal to 0.01 The %RSD must be less than or equal to 50%. The %RSD must be less than or equal to 30% for 2,4-Dinitrotoluene, 2-Nitrophenol, and 2,4-Dimethylphenol, and less than or equal to 20.5% for all other compounds and DMC’s.

<u>SEMIVOLATILE COMPOUNDS WITH POOR RESPONSE</u>

SEMIVOLATILE COMPOUNDS

2,2'oxybis(1-Chloropropane)	Benzaldehyde
4-Chloroaniline	Pentachlorophenol
Hexachlorobutadiene	4-Nitroaniline
Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol
2-nitroaniline	N-Nitrosodiphenylamine
3-nitroaniline	3,3'-Dichlorobenzidine
2,4-Dinitrophenol	4-Chloroaniline-d4 (DMC)
4-Nitrophenol	4,6-Dinitro-2-methylphenol-d2 (DMC)
Acetophenone	4-Nitrophenol-d4 (DMC)
Caprolactam	

12.2 Are response factors stable (%RSD # 20.5, # 50 for poor performers and # 30 for 2,4-Dinitrotoluene, 2-Nitrophenol, and 2,4-Dimethylphenol) for Semivolatiles over the entire concentration range of the calibration? [ ] ___ ___

ACTION: Circle all outliers in red.

NOTE: Although 24 Low Concentration semivolatile compounds have a minimum RRF and no maximum %RSD, the technical acceptance criteria are the same for all analytes.

ACTION: If the %RSD exceeds the above criteria, qualify positive results for that analyte "J" and non-detects using professional judgement. When %RSD > 90%, flag all non-detects for that analyte "R", and positive hits as “J”.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

12.3 Are any RRFs **<** 0.05, **<** 0.01 for poor performers? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: If any RRF < 0.05, or < 0.01 for poor performers:

1. Flag "R" all non-detects.

2. Flag "J" all positive results.

12.4 Are there any transcription/calculation errors in the reporting of, RRFs, RRFs or % RSD values? (Check at least two values but if errors are found, check more.) ___ [ ] ___

ACTION: If errors are large, take action as specified in section 3.1 above.

NOTE: Contract Requirements: The SOW allows up to four (see sec. 9.3.5.4, p. D-21/SV) of the required analytes to fail contractual %RSD or RRF criteria, provided the %RSD is # 40.0 and RRF is $ 0.010. (See Table D-4, page D-48, 49/SV and analytes marked with a "*" on Form VI LCSV for a list of required analytes and contractual criteria.

ACTION: If more than four analytes fail %RSD or RRF criteria, document in the Data Assessment under Contract Problems/Non-Compliance.

13.0 GC/MS Continuing Calibration (Form VII LCSV)

13.1 Are the Continuing Calibration Forms (Form VII LCSV-1 & -2) present and complete for the semivolatile fraction? [ ] ___ ___

13.2 Has a continuing calibration standard been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

ACTION: List below all sample analyses that were not within twelve hours of a continuing calibration analysis for each instrument used.

____

____

ACTION: If any forms are missing or no continuing calibration standard has been analyzed within twelve hours of

every sample analysis, notify the TOPO to obtain explanation/resubmittals. If continuing calibration data are not available, flag all associated sample data as unusable (R).

13.3 Do any semivolatile compounds have a %D between the initial RRF and continuing RRF which exceeds the ± 25.0% criteria? ___ [ ] ___

ACTION: Circle all outliers in red.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When %D is > 90%, reject all non-detects for that analyte (R) unusable and positive results "J".

13.4 Do any semivolatile compounds have a RRF < 0.05, <0.01 for the poor performers? ___ [ ] ___

ACTION: Circle all outliers with red pencil.

ACTION: If the RRF is < 0.05, < 0.01 for the poor performers, qualify associated positive results estimated (J) and non-detects unusable (R).

13.5 Are there any transcription/calculation errors in the reporting of continuing RRFs or %D between initial RRFs and continuing RRFs? (Check at least two values, but if errors are found check more.) ___ [ ] ___

ACTION: Circle errors with red pencil.

ACTION: If errors are large, notify the TOPO to obtain explanation/resubmittals, make any necessary corrections and document the effect in the data assessment.

14.0 Internal Standards (Form VIII LCSV)

14.1 Are the Internal Standard Area and RT Summary Forms (Form VIII LCSV-1 & -2) present and complete for the semivolatile fraction? [ ] ___ ___

YES NO N/A

14.2 Are the internal standard areas for every sample and blank within the upper and lower limits (-50% to +100%) for each continuing calibration? [ ] ___ ___

ACTION: Circle errors with red pencil.

ACTION: List all the outliers below.

Sample #	Internal Std	Area	Lower Limit	Upper Limit
________	____________	____________	____________	___________
________	____________	____________	____________	___________

ACTION: 1. If the internal standard area count is outside the upper or lower limit, flag all positive results and non-detects quantitated with this internal standard "J" and "UJ", respectively.

2. Do not qualify non-detects associated with IS areas > 100%.

3. If the IS area is < 50%, qualify all associated non-detects estimated “R”.

INTERNAL STANDARDS ACTIONS FOR SEMIVOLATILES

CRITERIA	ACTION	
	Detected Associated Compounds	Non-Detected Associated Compounds
Area counts > 100% of 12-hour standard	“J”	No Action
Area counts < 50% of 12-hour standard	“J”	“R”

14.3 Are the retention times of the internal standards within 20 seconds of the associated calibration standard? [ ] ___ ___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 20 seconds.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for Low Concentration semivolatile analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. If large differences exist, contact the TOPO to confirm identification of field duplicates with the sampler.

PART C: PESTICIDE/AROCLOR ANALYSIS

1.0 Sample Conditions/Problems

1.1 Do Traffic Reports/Chain-of-Custody records or SDG Narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? [ ] ___ ___

ACTION: If samples were not iced, or the ice was melted upon arrival to the laboratory, and the temperature of the cooler was > 10° C, flag all positive results "J" and all non-detects "UJ".

ACTION: Check extraction log for sample pH, if adjustment was needed, it should have been noted in the SDG Narrative. If more information is needed, notify the TOPO to contact the lab.

2.0 Holding Times

2.1 Have any Pest/Aroclor technical holding times, determined from date of collection to date of extraction, been exceeded? ___ [ ] ___

Technical Holding Times: Continuous liquid-liquid extraction of samples for Pesticide/Aroclor analysis must begin within seven days of collection. Extracts must be analyzed within 40 days of extraction.

Table of Holding Time Violations
(See Chain-of-Custody records)

Sample ID	Date Sampled	Date Lab Received	Date Extracted	Date Analyzed
________	________	________	________	________
________	________	________	________	________
________	________	________	________	________
________	________	________	________	________

_________ _________ _________ _________ _________

_________ _________ _________ _________ _________

ACTION: If technical holding times were exceeded, flag all positive results as estimated (J) and sample quantitation limits (UJ) and document in the Data Assessment that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all the data should at least be qualified "J", but the reviewer may determine that non-detects are unusable (R).

NOTE: Contractual Holding Times: Extraction of water samples must begin within 5 days VTSR. All laboratory extracts must be analyzed within 40 days of the VTSR.

ACTION: If contractual holding times were exceeded, document in the Data Assessment under Contract Problems/Non-Compliance.

3.0 Surrogate Recovery (Form II LCP)

3.1 Are the Low Concentration Semivolatile Surrogate Recovery Summaries (Form II LCSV) present and complete for all samples? [ ] ___ ___

ACTION: Notify the TOPO that explanation/resubmittals are required from the laboratory. If missing deliverables are unavailable, document effect in data assessments.

3.2 Were outliers marked correctly with an asterisk? [ ] ___ ___

ACTION: Circle all outliers with red pencil.

3.3 Were surrogate recoveries of TCX or DCB in any sample or blank outside of the contractual limits of 30 - 150%? ___ [ ] ___

ACTION: If either surrogate spike recovery is outside the acceptance limits, the Validator must consider the existence of coelution and interference in the raw data and use professional judgement as described below, as surrogate recovery problems may not directly apply to target analytes.

1. For any surrogate recovery greater than 150%:

   a. Qualify positive hits as estimated "J".

   b. Do not qualify Non-detects.

2. For any surrogate recovery greater than or equal to 10%, but less than 30%.

   a. Qualify positive hits as estimated "J".

   b. Qualify Non-detects as "UJ".

3. For any surrogate recovery less than 10%, ignoring dilutions, and in the absence of interference

   a. Qualify positive hits as estimated "J".

   b. Qualify Non-detects as unusable "R".

Surrogate Actions for Pest/PCB Analyses

Criteria	**Action** *	
	Detected Associated Compounds	**Non-detected Associated Compounds**
%R > 150%	"J"	No qualification
10% ≤ %R < 30%	"J"	"UJ"
%R < 10% (ignore dil's)	"J"	"R"
RT out of RT window	Professional Judgement	

***** Use professional judgement in qualifying data as surrogate recovery problems may not directly apply to target analytes.

Pesticides Surrogates and Associated Target Compounds

Tetrachloro-m-Xylene	**Decachlorobiphenyl**	
alpha-BHC	alpha-Chlordane	4,4'-DDE
beta-BHC	gamma-Chlordane	4,4'-DDT
gamma-BHC	Heptachlor epoxide	Endosulfan I
delta-BHC	Dieldrin	Endosulfan II
Heptachlor	Endrin	Endosulfan sulfate
Aldrin	Endrin Aldehyde	Methoxychlor
	Endrin ketone	Aroclors
	4,4'-DDD	Toxaphene

3.4 Were surrogate retention times (RT) within the windows established during the initial 3-point analysis of Individual Standard Mixture A (See Form VI LCP-1)? [ ] ___ ___

ACTION: If the RT limits are not met, positive results and non-detects may be qualified unusable (R) for that sample based on professional judgement.

3.5 Are there any transcription/calculation errors between raw data and Form II? ___ [ ] ___

ACTION: If large errors exist, notify the TOPO to obtain explanation/resubmittals. Make any necessary corrections and document effect in data assessments.

4.0 Laboratory Control Sample (LCS)

4.1 Is the Laboratory Control Sample (LCS) Recovery Form (Form III LCP-2) present? [ ] ___ ___

4.2 Was the LCS analyzed at the required frequency (once per SDG, or every 20 samples) for the Low Concentration Pest/Aroclor method? [ ] ___ ___

ACTION: If any LCS data are missing, take action as specified in 3.1 above.

4.3 How many PEST spike recoveries (see Table below) are outside QC limits listed in Table D-3, page D-61/PEST of the SOW?

Pesticides Laboratory Control Sample (LCS) spike compounds and limits.

LCS Spike Compound	Recovery Limits (%)	LCS Spike Compound	Recovery Limits (%)
gamma-BHC	50-120	Endosulfan sulfate	50-120
Heptachlor epoxide	50-150	gamma-Chlordane	30-130
Dieldrin	30-130	TMX (Surrogate)	30-150
4,4'-DDE	50-150	DCB (Surrogate)	30-150
Endrin	50-120		

ACTION: Check calculations, surrogates, LCS solutions and instrument performance.

ACTION: Qualify only the specific analytes included in the LCS solution in the following two situations:

1. If the LCS recovery is greater than the upper QC limit, qualify positive results for the affected

compound(s) estimated (J). Do not qualify non-detects.

2.If the LCS recovery is less than the lower QC limit, then qualify positive results for the affected compound(s) estimated (J) and non-detects unusable (R).

Qualify <u>all sample results</u> in the following situations

1. If 25% or more of the analyte recoveries are below QC limits qualify all associated positive results "J" and non-detects "R".

2. If two or more analytes exhibit < 10% recovery, qualify all associated positive results "J" and non-detects "R".

It should be noted in the Data assessment if a laboratory fails to analyze an LCS with each SDG, or consistently fails to generate acceptable LCS recoveries.

5.0 <u>Laboratory MS/MSD (Form III LCP-1)</u>

5.1 Is the Pest/PCB MS/MSD Recovery Form (Form III LCP-1) present? [ ] ___ ___

5.2 Was the MS/MSD analyzed at the required frequency (Once per SDG, or every 20 samples? [ ] ___ ___

ACTION: If any MS/MSD data are missing, take action as Specified in 3.1 above.

ACTION: No action is taken on MS/MSD <u>alone.</u> However, using professional judgement, the Validator may use the MS and MSD results in conjunction with other QC criteria and determine the need for some qualification of the data.

6.0 <u>Blanks (Form IV LCP)</u>

6.1 Is the Method Blank Summary (Form IV LCP) present? [ ] ___ ___

	YES	NO	N/A
6.2 Frequency of Analysis: For the analysis of Pesticide/Aroclor TCL compounds, has a method blank been analyzed concurrently for each SDG, every 20 samples or each extraction batch, whichever is more frequent?	[ ]	___	___

ACTION: If any blank data are missing, take action as specified in section 3.1 above. If blank data is unavailable, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

	YES	NO	N/A
6.3 A separate Form IV LCP should be present if just part of an extraction batch required sulfur removal. In such cases some samples will be listed on two blank summary forms - once under the method blank, and once under the sulfur clean-up blank (PCBLK). Was this additional blank raw data and Form IV LCP submitted when required?	[ ]	___	___

ACTION: If sulfur clean-up blank data and Form IV are missing, take action as specified in 3.1 above.

	YES	NO	N/A
6.4 Has a Pest/Aroclor instrument blank been analyzed at the beginning of every 12 hr. period following the initial calibration sequence (minimum contract requirement)?	[ ]	___	___

ACTION: If any blank data are missing, take action as specified in section 3.1 above.

	YES	NO	N/A
6.5 Was the correct identification scheme used for all Pest/PCB blanks? (See SOW, page B-30, section 3.3.7.3 for further details.)	[ ]	___	___

ACTION: Contact the TOPO to obtain resubmittals or make the required corrections on the forms. Document in the Data Assessment under Contract Problems/Non-Compliance all corrections made by the validator.

6.6 Chromatography: Review the blank raw data - chromatograms, quant reports or data system printouts. Is the chromatographic performance

	YES	NO	N/A
(baseline stability) for each instrument acceptable for Pest/PCBs?	[ ]	___	___

ACTION: Use professional judgement to determine the effect on the data.

7.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

	YES	NO	N/A
7.1 Do any method/instrument/cleanup blanks have positive results for Pest/Aroclors?	___	[ ]	___
7.2 If any method, instrument and/or sulfur clean-up blanks contain "hits" for target compounds, are these hits greater than the CRQL for that analyte?	___	[ ]	___

ACTION: Note in the Data Assessment under Contract Problems/Non-Compliance if any method, instrument or sulfur clean-up blank(s) contain hit(s) at concentration(s) greater than the CRQL for that analyte.

	YES	NO	N/A
7.3 Do any field/rinse blanks have positive Pest/Aroclor results?	___	[ ]	___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

NOTE: When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor.

Flag sample result with a "U":	**Report CRQL & qualify "U":**	**No qualification is needed:**
Sample conc. > CRQL, but < 1x blank.	Sample conc. < CRQL & is < 1x blank value.	Sample conc. > CRQL & > 1x blank value.

NOTE: If gross blank contamination exists, all data in the associated samples should be qualified as unusable (R).

7.4 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: Note in Data Assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 Calibration and GC Performance

8.1 Are the following gas chromatograms and data systems printouts for both columns present for all samples, blanks, and LCS:

a. Peak Resolution Check? [ ] ___ ___

b. PEM standards? [ ] ___ ___

c. Aroclor 1016/1260? [ ] ___ ___

d. Aroclors 1221, 1232, 1242, 1248, 1254? [ ] ___ ___

e. Toxaphene? [ ] ___ ___

f. Low points Individual Mixtures A & B? [ ] ___ ___

g. Med points Individual Mixtures A & B? [ ] ___ ___

	YES	NO	N/A
h. High points Individual Mixtures A & B?	[ ]	___	___
i. Instrument blanks?	[ ]	___	___
j. Were appropriate GC columns used (see SOW, page D-10/PEST, section 6.10.1.3)?	[ ]	___	___

ACTION: If no, take action as specified in 3.1 above.

	YES	NO	N/A
8.2 Do chromatograms for all initial calibration standards (Resolution Check Mixtures, Individual Standard Mixtures A & B and PEM) display single component peaks at > 10% but < 100% of full scale?	[ ]	___	___
Do chromatograms for multi-component standards display all peaks between 25% and 100% of full scale?	[ ]	___	___
Were chromatograms for at least one each of Standard Mixtures A & B replotted to display standard peaks between 50% and 100% of full scale?	[ ]	___	___
Have chromatograms for the above standards been replotted, when necessary, showing the scaling factor used to meet the above requirements?	[ ]	___	___

NOTE: All standard chromatograms must clearly display single component peaks at > 10% but < 100% of full scale, and multi-component peaks between 25% and 100% of full scale. At least one analysis each of Standard Mixtures A & B must display standard peaks between 50% and 100% of full scale. Chromatograms must be replotted, if necessary, to accommodate peaks not properly scaled initially. Both the initial and replotted chromatograms must be submitted with the data package. (See SOW, page D-25/PEST, section 9.2.5.10 for details.)

ACTION: If all single component peaks in all standard chromatograms are not clearly displayed and properly scaled, notify the TOPO to obtain resubmittals of the necessary data.

8.3 Are Forms VI LCP-1 through VI LCP-7 present and complete for each column and each analytical sequence? [ ] ___ ___

ACTION: If no, take action specified in 3.1 above.

8.4 Are there any transcription/calculation errors between raw data and Forms VI LCP? ___ [ ] ___

ACTION: If large errors exist, notify the TOPO to obtain explanation/resubmittals, make necessary corrections and document the effect in data assessments.

8.5 Do all standard retention times, for each pesticide in each level of Individual Mixtures A & B, fall within the windows established during the initial calibration sequence (see Form VI LCP-1)? [ ] ___ ___

ACTION: If no, all samples in the entire analytical sequence are potentially affected. Check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R). For Aroclors, the RT may be outside the RT window (Form VI LCP-3), but the Aroclor may still be identified from the individual pattern.

8.6 Have the linearity criteria been satisfied for the initial analyses of Individual Standard Mixtures A & B for both columns (Form VI LCP-2)? %RSD must be # 25.0 for "- and *-BHC, # 30.0 for the two surrogates and # 20.0 for all other analytes. [ ] ___ ___

NOTE: Contractual requirements allow up to two single-component analytes, except surrogates, to exceed the linearity criteria provided %RSD # 30.0. (See SOW, section 9.2.5.7, page D-25/PEST.) The technical criteria, however, are the same for all analytes.

ACTION: If technical criteria were not met, qualify all associated positive results generated during the entire analytical sequence "J" and all non-detects "UJ". If %RSD is > 90, flag all non-detects for that analyte unusable (R).

ACTION: Note in the Contract Problems/Non-Compliance section of the Data Assessment and the Organic Regional Data Assessment Summary if more than two analytes exceeded the 20.0 percent limit.

8.7 Is the resolution between each pair of adjacent peaks in the Resolution Check Mixture $ 60.0% on both columns (Form VI LCP-4)? [ ] ___ ___

ACTION: If no, qualify positive results for inadequately resolved compounds "J". Use professional judgement to determine if non-detects, which elute in areas affected by coeluting peaks, should be qualified "N" (presumptive evidence of presence) or "R" (unusable).

8.8 Is Form VI LCP-5 present and complete for each PEM standard used for <u>both initial and continuing</u> calibrations (see SOW page B-45, section 3.12.4)? [ ] ___ ___

ACTION: If no, take action as specified in section 3.1 above.

8.9 For each PEM standard, was the resolution between each pair of adjacent peaks $ 90.0% on both columns? [ ] ___ ___

ACTION: Qualify positive results for compounds not adequately resolved estimated (J). Qualify non-detects based on professional judgement.

8.10 Have Forms VI LCP-6 & -7 been completed for all midpoint Individual Standards A and B used for initial calibration? [ ] ___ ___

For each standard, was the resolution between each pair of adjacent peaks $ 90.0% on both columns? [ ] ___ ___

ACTION: If no, qualify positive results for compounds that were not adequately resolved estimated (J). Use

professional judgement to determine if non-detects which elute in areas affected by co-eluting peaks should be qualified "N" (presumptive evidence of presence) or unusable (R).

8.11 Is Form VII Pest-1 present and complete for each PEM standard analyzed during the analytical sequence for both columns? [ ] ___ ___

Was the % breakdown of DDT and Endrin calculated using the equations given on page D-22/PEST, sec. 9.2.4.8 in the SOW? [ ] ___ ___

Were all pesticides and surrogates in each PEM standard within the RT windows established during the Initial Calibration? [ ] ___ ___

ACTION: If no, take action as specified in section 3.1 above.

8.12 Has the individual % breakdown on either column exceeded 20.0% for:

4,4'-DDT? ___ [ ] ___

Endrin? ___ [ ] ___

Has the combined breakdown for 4,4'-DDT and Endrin exceeded 30.0% on either column (required for all PEM analyses)? ___ [ ] ___

ACTION: 1. If any % breakdown has failed the QC criteria in either PEM in steps 2 and 17 in the <u>initial calibration</u> sequence (SOW, page D-20/PEST, section 9.2.3.4) qualify all sample analyses in the entire analytical sequence as described below.

2. If any % breakdown has failed the QC criteria in a PEM Verification calibration, review data beginning with the samples which followed the last <u>in-control</u> standard until the next acceptable PEM & qualify the data as described below.

a. <u>4,4'-DDT Breakdown</u>: If 4,4'-DDT breakdown is greater than 20.0%:

i. Qualify all positive results for 4,4'-DDT "J".

ii. Qualify positive results for 4,4'-DDD and/or 4,4'-DDE "J".

iii. If 4,4'-DDT was not detected, but 4,4'-DDD and/or 4,4'-DDE are detected qualify the quantitation limit for 4,4'-DDT as unusable "R", and qualify positive results for 4,4'-DDD and/or 4,4'-DDE as presumptively present at an approximated quantity "NJ".

b. Endrin Breakdown: If Endrin breakdown is greater than 20.0%:

i. Qualify all positive results for Endrin with "J".

ii. Qualify positive results for Endrin ketone and Endrin aldehyde as estimated "J".

iii. If Endrin was not detected, but Endrin Aldehyde and/or Endrin ketone are detected, qualify the quantitation limit for Endrin as unusable "R", and qualify positive results for Endrin Aldehyde and/or Endrin ketone as presumptively present at an approximate quantity "NJ".

c. Combined Breakdown: If the combined 4,4'-DDT and Endrin breakdown is greater than 30.0%:

i. The validator should consider the degree of individual breakdown of 4,4'-DDT and Endrin and apply qualifiers as described above.

8.13 Are the %D values for all PEM analytes $ -25.0% and # +25.0% (Form VII LCP-1)? [ ] ___ ___

ACTION: If no, qualify all associated positive results generated during the analytical sequence "J" and sample quantitation limits "UJ".

NOTE: If the failing PEM is part of the initial calibration, all samples are potentially affected. If the offending standard is a verification calibration, the associated

samples are those which followed the last <u>in-control</u> standard until the next passing standard.

8.14 Have all samples been injected within 12 hrs. of an acceptable instrument blank? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity to the effect on data reliability.

8.15 Is Form VII LCP-2 present and complete for each INDA and INDB calibration verification analyzed? [ ] ___ ___

ACTION: If no, take action as specified in section 3.1 above.

8.16 Are there any transcription/calculation errors between raw data and Form VII LCP-2? ___ [ ] ___

ACTION: If large errors exists, notify the TOPO that explanation/resubmittals from the lab are required. Make any necessary corrections and document in the Data Assessment under Contract Problems/Non-Compliance.

8.17 Do all standard retention times for each INDA and INDB Verification Calibration fall within the windows established during the initial calibration sequence? [ ] ___ ___

ACTION: If no, beginning with the samples which followed the <u>last in-control standard</u>, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

8.18 Are all %D values for INDA and INDB calibration verification compounds $ -25.0% and # +25.0%? [ ] ___ ___

ACTION: If the %D is outside the ±25.0% range for any compound(s), qualify associated positive results for that compound "J" and non-detects "UJ". The "associated samples" are those which followed the <u>last</u>

in-control standard up to the next passing standard containing the analyte(s) in question. If the %D is > 90%, flag all non-detects for that analyte "R" (unusable).

9.0 Analytical Sequence Check (Form VIII LCP)

9.1 Is Form VIII LCP present and complete for each column and each period of analyses? [ ] ___ ___

ACTION: If no, take action specified in 3.1 above.

9.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses (see SOW pages D-39 & D-40/PEST)? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

9.3 Were all samples analyzed within a 12 hour time period beginning with the injection of an instrument blank and bracketed by acceptable analyses of the proper standards? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify accordingly. Document in the Data Assessment under Contract Problems/Non-Compliance.

9.4 If a multi-component analyte was detected in a sample, was a matching multi-component standard (Toxaphene or Aroclors) analyzed within 72 hours of the sample and within a valid 72-hr. sequence? [ ] ___ ___

NOTE: This standard is for identification purposes only. Positive results for Aroclors and Toxaphene are quantitated from the initial calibration.

ACTION: If no, document in the Contract Problems/Non-Compliance section of the Data Assessment and Organic Regional Data Assessment Summary.

10.0 Cleanup Efficiency Verification (Form IX LCP)

10.1 Is Form IX LCP present and complete for each lot of Florisil Cartridges used? (Florisil cleanup is required for all Pest/Aroclor extracts.) [ ] ___ ___

Are all samples listed on the Pesticide Florisil Cartridge Check Form? [ ] ___ ___

ACTION: If no, take action specified in 3.1 above. If the data suggests Florisil cleanup was not performed, note in the Data Assessment under Contract Problems/Non-Compliance.

10.2 Are percent recoveries (% REC) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedure within QC limits, 80 - 120%, for the Florisil cartridge check? [ ] ___ ___

ACTION: If %REC of one or two TCL compounds is < 80%, qualify positive results "J" and non-detects "UJ" for these compounds.

If more than two compounds exhibited < 80% recovery, qualify all associated positive results "J" and non-detects "UJ".

If two or more have %REC < 10%, qualify all positive results "J", and non-detects "R". Use professional judgement to qualify positive results if recoveries are > 120%.

NOTE: Sample data should be evaluated for potential interferences if recovery of 2,4,5-Trichlorophenol was > 5% in the Florisil Cartridge Performance Check analysis. Note in Contract Problems/Non-Compliance section of reviewer narrative.

11.0 Pesticide/Aroclor Identification (Forms X LCP-1 & -2)

11.1 Are Forms X LCP complete for every sample in which a pesticide and/or Aroclor were detected? [ ] ___ ___

ACTION: If no, take action specified in 3.1 above.

11.2 Are all sample chromatograms properly scaled, attenuated, etc. as required for proper identification of single and multi-component analytes? (See SOW, page D-46/PEST, sections 11.3.1 thru 11.3.9.8 for specific details.) ___ [ ] ___

NOTE: Proper verification of Pest/PCB results depends on clear, legible presentation of the raw data. Single component pesticides and all peaks chosen for quantitation of multi-component analytes must appear at less than 100% of full scale (see SOW). Toxaphene and PCB patterns must be clearly visible to enable comparison with standard chromatograms.

ACTION: If retention times or apex of peaks cannot be verified, or if multi-component peak patterns cannot be discerned, contact the TOPO to obtain rescaled chromatograms from the lab.

11.3 Are there any transcription/calculation errors between raw data and Forms 10LCA and 10LCB? [ ] ___ ___

ACTION: If large errors exists, notify the TOPO that explanation/resubmittals from the lab are required. Make any necessary corrections and document in the Data Assessment under Contract Problems/Non-Compliance and in the Organic Regional Data Assessment Summary.

11.4 Are retention times (RT) of sample compounds within the established RT windows for both analyses? [ ] ___ ___

ACTION: Use professional judgement to qualify positive results. Qualify as unusable (R) all positive results which were not confirmed on a second GC column. Also qualify as unusable (R) all positive results not within the RT window unless associated standards are similarly biased (see Functional Guidelines). Use professional judgement to assign an appropriate quantitation limit.

11.5 Is the percent %D calculated for positive sample results on the two columns > 25.0? ___ [ ] ___

NOTE: If %D is > 25.0, lab should have reported results with the "P" qualifier.

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be flagged as follows:

<u>% Difference</u>	<u>Qualifier</u>
0 - 25%	None
26 - 70%	"J"
71 - 100%	"JN"
> 100%	"R"
100 - 200% (Interference detected)*	"JN"
> 50% (Pesticide value is < CRQL)**	"U"

* When the reported %D is 100 - 200%, but interference is suspected on either column, qualify the data with "J".

** When the **<u>reported</u>** <u>pesticide value</u> is lower than the CRQL, and the %D is > 50%, raise the value to the CRQL and qualify "U", undetected.

NOTE: For Aroclors, if the %D is > 50%, but the pattern of GC peaks on both columns indicates a specific Aroclor is present, qualify that Aroclor "J".

NOTE: The lower of the two values is reported on Form I. If using professional judgement, the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the Data Assessment.

11.6 Check chromatograms for false negatives (especially the multiple peak compounds Toxaphene and PCBs). Were there any false negatives? ___ [ ] ___

ACTION: Use professional judgement to decide if the compound should be reported. If the appropriate Aroclor standards were not analyzed within 72 hrs. of the sample(s) in question, qualify the data unusable (R).

Also note in Data Assessment under Contract Problems/Non-Compliance if the lab failed to analyze Aroclor standards when required.

12.0 Target Compound List

12.1 Are the Organic Analysis Data Sheets (Form 1 LCP) present with required header information for each of the following:

	YES	NO	N/A
a. Samples?	[ ]	___	___
b. LCS analyses?	[ ]	___	___
c. Method Blanks?	[ ]	___	___
d. Instrument Blanks?	[ ]	___	___
e. Matrix Spike/Matrix Spike Duplicate?	[ ]	___	___

12.2 Are the chromatograms and quant. reports included in the sample data package for each of the following:

	YES	NO	N/A
a. Samples?	[ ]	___	___
b. LCS analyses?	[ ]	___	___
c. Method Blanks?	[ ]	___	___
d. Instrument Blanks?	[ ]	___	___
e. Matrix Spike/Matrix Spike Duplicate?	[ ]	___	___

ACTION: If any data are missing, take action as specified in section 3.1 above.

12.3 Is chromatographic performance acceptable with respect to:

	YES	NO	N/A
a. Baseline stability?	[ ]	___	___
b. Resolution?	[ ]	___	___
c. Peak shape?	[ ]	___	___
d. Full-scale graph attenuation?	[ ]	___	___

	YES	NO	N/A
e. Other: ________________________________?	[ ]	___	___
12.4 Were any electropositive displacement (negative peaks) or unusual peaks seen?	___	[ ]	___

ACTION: Use professional judgement to determine the acceptability of the data. Address comments under System Performance section of the Data Assessment.

13.0 Compound Quantitation and Reported Detection Limits

	YES	NO	N/A
13.1 Are there any transcription/calculation errors in Form I results? Check at least two positive results. Were any errors found?	___	[ ]	___

NOTE: Single-peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. Use professional judgement to decide whether a large discrepancy indicates the presence of an interfering compound. If an interfering compound is suspected, the lower of the two values should be reported and qualified as presumptively present at an approximated quantity "JN". This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has interfered with the evaluation of the second column confirmation.

	YES	NO	N/A
13.2 Are the CRQLs adjusted to reflect sample dilutions?	[ ]	___	___

ACTION: If large errors exist, take action as specified in section 3.1 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQLs from the diluted sample). Replace concentrations which exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with the result from the diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including those in the data summary package.

ACTION: Quantitation limits affected by large, off-scale peaks should be qualified as unusable (R). If the interference is on-scale, the reviewer may offer an approximated quantitation limit (UJ) for each affected compound.

NOTE: If a sample required greater than a 10 times dilution, then a 10 times more concentrated analysis must also be performed and submitted (see SOW, page D-41/PEST, section 10.2.**3.5**).

ACTION: If a more concentrated analysis is unavailable, document in the Contract Problems/Non-Compliance section of the Data Assessment. Use professional judgement to qualify non-detects and positive hits below the CRQL.

14.0 Field Duplicates

14.1 Were any field duplicates submitted for Pest/Aroclor analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. If large differences exist, contact the TOPO to confirm identification of field duplicates with the sampler.

Definitions

BFB - bromofluorobenzene
BHC - benzene hexachloride
BNA - base neutral acid
CADRE - Computer Aided Data Review and Evaluation
CARD - CLP Analytical Results Database
CCS - contract compliance screening
CLASS - Contract Laboratory Analytical Services Support
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
DCB -decachlorobiphenyl
DDD - dichlorodiphenyldichloroethane
DDE - dichlorodiphenylethane
DDT - dichlorodiphenyltrichloroethane
GC - gas chromatography
GC/EC - gas chromatography/electron capture detector
GC/MS - gas chromatography/mass spectroscopy
GPC - gel permeation chromatography
kg - kilogram
:g - microgram
MAGIC - Mainframe Access Graphical Interface with CARD
R - liter
LCS - Laboratory Control Sample
LES - Laboratory Evaluation Sample
mR - milliliter
PCB - Polychlorinated Biphenyl
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
RRF - average relative response factor (from initial calibration)
RRT - relative retention time
RSD - relative standard deviation
RT - retention time
RSCC - Regional Sample Control Center
SDG - sample delivery group
SMC - system monitoring compound
SOP - standard operating procedure
SOW - Statement of Work
SVOA - semivolatile organic acid
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TCX -tetrachloro-m-xylene
TIC - tentatively identified compound
TPO - technical project officer
VOA - volatile organic acid

VTSR - validated time of sample receipt
TOPO - Task Order Project Officer

References

SOW/CLP OLC03.2
National Functional Guidelines (June 2001)